



Role of zirconia filler on friction and dry sliding wear behaviour of bismaleimide nanocomposites

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ABSTRACT

This paper discusses the friction and dry sliding wear behaviour of nano-zirconia (nano-ZrO₂) filled bismaleimide (BMI) composites. Nano-ZrO₂ filled BMI composites, containing 0.5, 1, 5 and 10 wt.% were prepared using high shear mixer. The influence of these particles on the microhardness, friction and dry sliding wear behaviour were measured with microhardness tester and pin-on-disc wear apparatus. The experimental results indicated that the frictional coefficient and specific wear rate of BMI can be reduced at rather low concentration of nano-ZrO₂. The lowest specific wear rate of 4×10^{-6} mm³/Nm was observed for 5 wt.% nano-ZrO₂ filled composite which is decreased by 78% as compared to the neat BMI. The incorporation of nano-ZrO₂ particles leads to an increased hardness of BMI and wear performance of the composites shows good correlation with the hardness up to 5 wt.% of filler loading. The results have been supplemented with scanning electron micrographs to help understand the possible wear mechanisms.

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1. Introduction

In recent years, nanoparticles have been used as fillers in polymeric composites for improving the tribological performance of the materials (nano-Al₂O₃/polyphenylene sulfide (PPS), nano-ZnO/polytetrafluoroethylene (PTFE), nano-TiO₂/epoxy, nano-SiO₂/polyarylate, nano-ZrO₂/polyetheretherketone (PEEK), nano-Si₃N₄/bismaleimide). Besides, fiber reinforcement is a traditional way to produce high performance composites materials. When nanoparticles are incorporated, both microstructure and properties of the composites can be optimized. Zhang et al. [1,2] systematically studied the wear resistance of epoxy filled with short carbon fiber, graphite, PTFE and nano-TiO₂ under different sliding conditions. The spherical nano-TiO₂ was able to apparently reduce the friction coefficient during sliding and consequently to reduce the shear stress, contact temperature and wear rate of fiber reinforced epoxy composites. Based on microscopic observations of the worn surfaces, a positive rolling effect of the nanoparticles between the materials pair was proposed, which led to remarkable reduction of friction coefficient. In order to improve the friction and wear behaviour of polymeric materials, one typical concept is to reduce their adhesion to the counterpart material and to enhance their

mechanical properties. This can be achieved quite successfully by using inorganic fillers. Ng et al. [3] verified that nanoparticles can remarkably reduce the wear rate, while micron sized particles cannot. Rong et al. [4] conformed that the dispersion state of the nanoparticles and micro-structural homogeneity of the fillers improved the wear resistance significantly. The way of nanoparticle incorporation must be considered as a very important key point to receive the desired material properties.

The composites containing nanoparticles exhibit the best anti-wear property by forming a uniform and tenacious transfer film on the ring surface. Especially, for PEEK/ZrO₂ nanocomposites. Zhang et al. [5,6] concluded that the addition of the grafted nanosilica into epoxy significantly reduced wear rate and frictional coefficient of the matrix at low filler loading. Compared with the cases of microsized silica and untreated nanosilica, the employment of grafted nanosilica provided the composites with much higher tribological performance. Wang et al. [7] presented experimental results which indicate that the ability of nano-ZrO₂ to improve the tribological behaviour is increased with decreasing particle size of the filler. The wear rate of larger particle size was high because the composite transfer film had poor adhesion to the counterface. On the other hand, the wear-resistant PEEK composite which was filled with small size ZrO₂ particles transferred well to the counterface and its transfer film was thin, uniform and adhered strongly to the counterface [1,2,7]. In the same way, the bismaleimide nanocomposites with surface-modified SiO₂ nanoparticles showed

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better wear resistance and lower frictional coefficient than composites with the unmodified nanoparticles SiO_2 [8]. The chemical treatment of the particle surface may further enhance the composite properties by improving the filler–matrix coupling quality [9]. For purposes of maximizing the use of nanoparticles, hybrid particulate fillers were employed in epoxy nanocomposites. In the work by Wetzel et al. [10], various amounts of micro- and nanoscale particles (calcium silicate CaSiO_3 , 4–15 μm ; alumina Al_2O_3 , 13 nm) were introduced into epoxy. Choosing the nanocomposite with the highest performance as a matrix, conventional CaSiO_3 micro-particles were further added in order to achieve additional enhancements in the properties. As a result, synergistic effects were found in the form of a further increase in wear resistance and stiffness.

One of the distinct advantages of nanocomposites over micro-composites lies in that the performance improvement is often acquired at relatively low concentration of the nano-fillers. Polymer composites must withstand high mechanical loads; it is usually reinforced with fillers. The addition of different fillers favorably stiffens the material and may also increase the strength under certain load conditions. However, some micro-fillers have a detrimental effect on the abrasion resistance [11,12].

In fact, it was verified experimentally by several research groups all over the world [9–13] that nanoparticles of metallic or inorganic type prove the ability to reinforce effectively thermoplastic and also thermosetting polymer matrices. Specifically, the reinforcement covers improvements of the flexural modulus without losing flexural strength. This effect is at the same time accompanied by improvements in fracture toughness and impact energy which, however, depend strongly on the filler volume content [14]. Only the nanofiller induced micromechanical deformation processes can be responsible for this behaviour [15]. However, the nanocomposite can only be effective, if the nanoparticles are well dispersed in the surrounding polymer matrix. It has been shown, that a considerable improvement in the mechanical and tribological properties can be achieved at very low filler volume content (<10%). The method of addition of nanoparticles must be considered as an important key point to get the desired material properties.

Bismaleimide (BMI) resin systems are derived from a variety of different starting chemical compounds, with some commonality in the final resin (Fig. 1). In the formulas, 4 and 4' denote the position of substituted maleimide group in the first and second benzene rings respectively. o and o' denote the position of allyl group, ortho (o) with respect to the –OH group in the first and second benzene

rings respectively. This BMI is synthesized from 4,4'-bismaleimidodiphenylmethane and maleic acid anhydride, with the synthesis being followed by cyclodehydration. It is the main building block of almost all commercially available BMI resins. BMI was synthesized according to the method for synthesis of allyl ether novlak described in [16]. These resin systems exhibit a high glass transition temperature (T_g) relative to post cure temperature, dimensionally stable at elevated temperatures (>260 °C) and have low flammability characteristics. In addition, they handle well in processing as a hot melt, exhibit good humidity resistance, good toughness and excellent mechanical properties at both ambient and elevated temperatures. Many modified BMI resin systems have been developed. Among them a two-component high performance resin system based on BDM and o,o'-diallyl bisphenol A (DBA), coded as BDM/DBA has been proved to have outstanding toughness, good humidity resistance, excellent thermal and mechanical properties [17]. Hence, the BDM/DBA resin was chosen as the base resin in the present work.

Nanocomposites using thermoplastic polymers are well known for improving the mechanical, electrical, thermal and insulating properties. However, nanocomposites using thermosetting polymers have not been studied extensively. Lin et al. [18] investigated the effect of interfacial adhesion between Kevlar-fiber and BMI matrix. Liu et al. [19] studied the micro-tribological behaviour of BMI/carbon nanotubes (CNTs) nanocomposite. Although, a good amount of work has been reported on the sliding wear behaviour of particulate filled polymer matrix composites as discussed earlier in this section, no literature could be cited on the sliding wear aspect of ZrO_2 filled BMI nanocomposites.

In this study, we have developed a new type of ZrO_2 nanoparticles filled BMI composites. A high shear mixing procedure was used to uniformly disperse the ZrO_2 nanoparticles into the BMI resin system. Effect of incorporation of ZrO_2 nanoparticles on microstructure, hardness, friction and dry sliding wear has been determined and analyzed.

2. Experimental procedure

2.1. Materials

4,4'-Bismaleimidodiphenylmethane (BDM) and o,o'-diallyl-bisphenol A (BA) were supplied by ABR Organics Limited, Hyderabad (India), Zirconia (ZrO_2) nanoparticles were purchased from Sigma Aldrich, Bangalore (India).

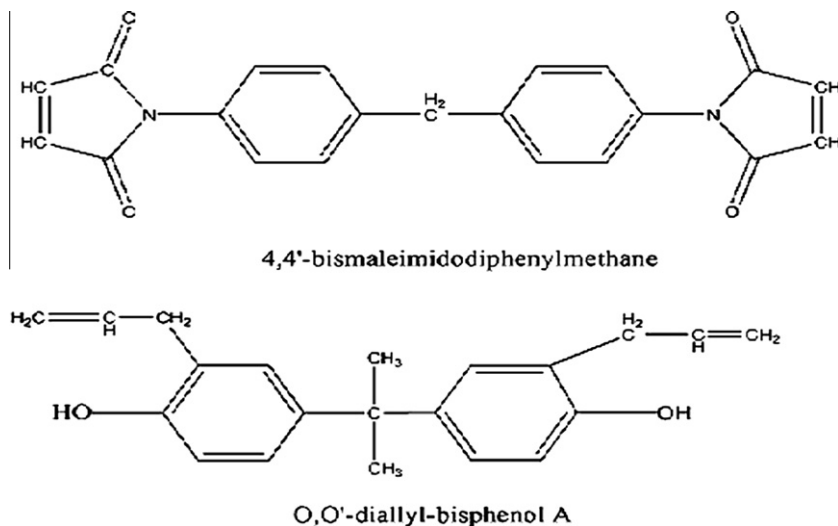


Fig. 1. Structural formulas. Top: 4,4'-bismaleimidodiphenylmethane (BDM); Bottom: o,o'-diallyl-bisphenol A (BA).

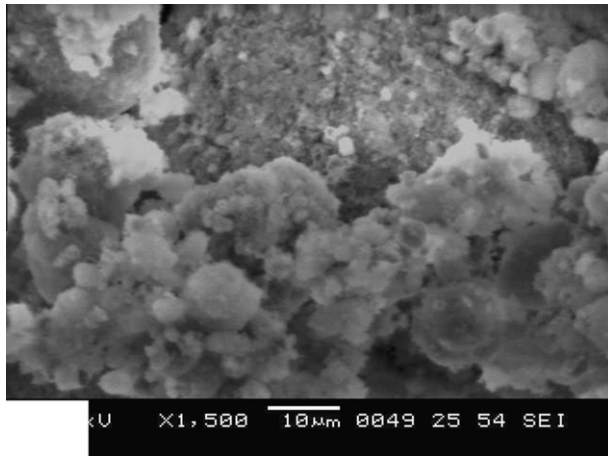


Fig. 2. Microstructure of zirconia nanoparticles.

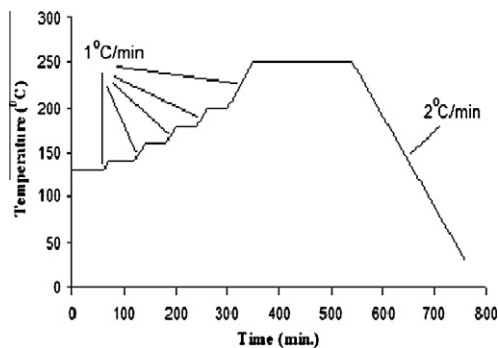


Fig. 3a. Curing cycle.

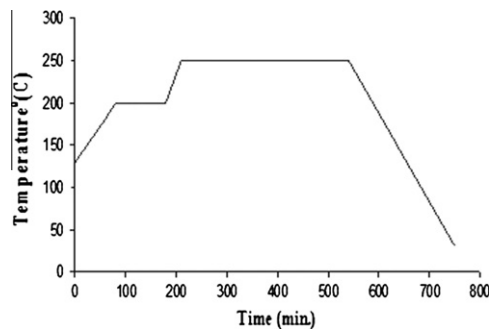


Fig. 3b. Post curing cycle (oven temperature).

The zirconia represents the ceramic nanocrystalline phase and consists of primary particles in the size range of 60–100 nm (Fig. 2). Along with a spherical shape, their large number is characterized by a very high specific surface area of 100 m²/g. The nanoparticles are provided as a dry powder. This powder contains particle agglomerates with sizes in the micrometer range (Fig. 2), which consist of ZrO₂ primary nanoparticles sticking strongly together. Primary nanoparticles attract each other due to adhesive inter-particle 'van der Waals' forces, which originate from the materials' surface energy.

2.2. Preparation of ZrO₂ filled BMI nanocomposites

Appropriate content of zirconia nanoparticles were added into BA at room temperature (25 °C) with vigorous mechanical agitating. The mixture was agitated for 2 h followed by ultrasonic

stirring for another 2 h to obtain a homogeneous suspension. Then the suspension was added into BDM at 130 °C with thorough stirring, and kept at that temperature for 15 min. After that the resultant mixture was poured into a preheated mold and degassed in a vacuum oven at 130 °C for 1 h. The mixture was cured in the oven as per the oven temperature profile in Fig. 3a. After curing, the panels were removed from the molds. Then the panels were again put in between the glass plates and clamped together. No rubber o-ring was used this time to eliminate the possibility of warp of the panel during post curing. The panels were again put back into the oven for post curing. The post cure cycle is shown in Fig. 3b. After post curing, the glass plates were removed and the panels were put in the desiccator for storage before use.

2.3. Characterization

The microhardness of samples was determined by conducting five replicated measurements on an automatic Vickers Microhardness tester (Model: MVH-I, Meta Tech Industries, Pune, India) at a load of 50 N and a loading time of 20 s. The average value of the five replicated measurements of hardness is adopted as the microhardness value of the specimens.

Unlubricated pin-on-disc sliding wear tests were carried out in order to determine the tribological properties of the nanocomposites. The disc material is made up of En-32 steel (diameter 160 mm and 8 mm thickness) having hardness value of HRC 65. The surface roughness of the disc varies from 0.02 to 0.06 μm. A constant 80 mm track diameter was used throughout the experimental work. The counterface slid against the BMI composite sample. Sliding was performed in air with the ambient temperature of around 25 °C over a period of 1 h at a sliding velocity of 0.84 m/s and a normal load of 40 N. Prior to wear testing, all specimens were cleaned, that is, the sample was abraded with water-abrasive paper (600 grit), a super-fine water-abrasive paper, and liquid diamond polishing agent, successively. Then both the steel ring and the specimen were cleaned with acetone and distilled water. After that the steel ring and the specimen were dried at 70 °C. The friction force was measured using a torque shaft equipped with strain gauges, and the friction coefficient was calculated automatically by taking into account the normal load and the friction force. The wear process takes some material away from the sample. This mass loss can accurately be measured by determining the weight of the specimen before and after the experiment. A characteristic value, which describes the wear performance under the chosen conditions for a tribo-system is the specific wear rate (K_s):

$$K_s = \frac{\Delta m}{\rho \cdot F_N \cdot L} \left(\frac{\text{mm}^3}{\text{Nm}} \right) \quad (1)$$

where Δm is the mass loss, ρ is the measured density of the composite, F_N is the normal load applied, and L is the sliding distance. In order to take repeatability into account, results from the friction and wear tests were obtained from three readings and the average value was adapted in our results.

Scanning electron microscopy examinations on a Jeol-5400 was used to study the morphology of worn surfaces. Before observation, the surface of the sample was sprayed with an even thin film of gold powder to improve the conductivity of the surface. The aim was to get information about the influence of fillers on the corresponding wear mechanisms.

3. Results and discussion

3.1. Effects of concentrations of ZrO₂ on the microhardness

Generally, the microhardness of a material has close relationship with the tribological behaviour of the material. So it is necessary to

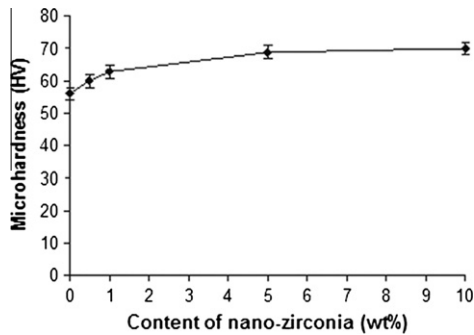


Fig. 4. Microhardness of neat BMI and nano-zirconia filled BMI composites.

study the effects of content of ZrO_2 nanoparticles on the microhardness of the nanocomposites. As shown in Fig. 4, the H_v microhardness readings increased all the way from 57 of the neat BMI polymer to 68 in the 10 wt.% of nano- ZrO_2 filled BMI composites, implying a maximum increment of 19%. It has been shown that a composite with a higher hardness value will be accompanied with lower wear rate and friction coefficient [20]. Note that the hardness increment is more significant up to 5 wt.% of filler loading. The higher H_v for 5 wt.% filled BMI composite is thought to result partly from the intrinsic hardness of ZrO_2 and these nano particles might impose better resistance against BMI segment motion under indentation. However, further loading of filler from 5 to 10 wt.%, the increase in hardness is very marginal suggesting that higher filler loading gives rise to poor dispersion and agglomeration in the BMI matrix. Further, it might not be able to elaborate their full strengthening capability in hardness improvement. It seems that the 5 wt.% nano- ZrO_2 particles could be more uniformly distributed and contributed the continuous hardness improvement.

3.2. Effects of concentration of ZrO_2 on the specific wear rate and friction coefficient

In general, the friction and wear properties do always describe the whole tribological system rather than a material property alone. Fig. 5 shows the specific wear rate (K_s) of the nanocomposites as a function of nano- ZrO_2 particle content. It was found that the nano- ZrO_2 filled BMI composite exhibited a decreased K_s in comparison to the neat BMI. However, the K_s of the composites were not linearly related to the content of nano- ZrO_2 content. The K_s sharply decreased when nano- ZrO_2 content was below 1 wt.%. With the nano- ZrO_2 content between 1 and 5 wt.%, the K_s decreased slightly with the increasing nano- ZrO_2 content. The lowest specific wear rate was obtained with the nano- ZrO_2 -BMI composite containing 5 wt.%. Although, the K_s increased with increasing nano- ZrO_2 above 5 wt.%, it was still lower in comparison

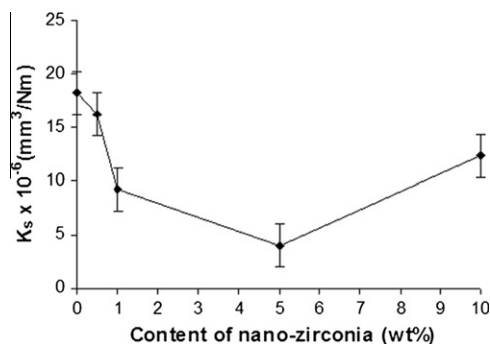


Fig. 5. Specific wear rate of neat BMI and nano-zirconia filled BMI nanocomposites.

to neat BMI. Among the filled nanocomposites, 5 wt.% nano- ZrO_2 filled BMI showed the lowest specific wear rate. Rong et al. [4] examined the influence of microstructure on the tribological performance of nanocomposites by different compounding methods. They conformed that the dispersion state of the nanoparticles and micro-structural homogeneity of the fillers improve the wear resistance significantly. When the micro-structural homogeneity of the nanocomposites was improved, their wear resistance could be increased significantly. The phenomenon reported in the present work is similar to what is reported in Ref. [4]. However, a further increase in filler loading leads obviously to a deterioration of the specific wear rate. A change in wear mechanism could be involved which may be ascribed to the large amount of hard ceramic nano- ZrO_2 particles causing a higher abrasive wear. Some aspects of the reinforcing role that ceramic microparticles can play in the tribological behaviour were studied by Durand [21], who found that large particles protect the matrix better than smaller ones because they can shield the polymer if they are not pulled out. Small particles, on the other hand, were removed and then involved in an abrading wear process.

Some important characteristics of nanocomposites have to be considered in order to explain this phenomenon. The quality of the interface in nanocomposites, usually play a very vital role in the materials capability to transfer stresses and elastic deformation from the matrix to the fillers [22]. If the filler matrix interaction is poor, the nanoparticles are unable to carry any part of the applied load. In that case, the wear resistance cannot be greater than that of neat polymer matrix. If the bonding between the polymer matrix and fillers is instead strong enough, the yield strength of the filled composite can be higher than that of the neat matrix [23]. In the same way, the difference in reducing the friction coefficient and improving the wear resistance ability of neat BMI and nano- ZrO_2 filled BMI composites should result mainly from the adhesion strength of the filler-matrix. Based on the experience from various studies, Friedrich et al. [24] concluded that fine particles contribute better to the property improvement under sliding wear conditions than larger particles. A similar trend was confirmed by Xing and Li [25] recently on the spherical silica particles filled epoxy when particle size varied from 120 to 510 nm. In the present work, the results are showing the similar trend.

Besides improving the wear resistance, the nanoparticles also reduce the coefficient of friction as shown in Fig. 6. Evidently, the nanocomposites with 5 wt.% nano- ZrO_2 filled BMI has the lowest friction coefficient. The coefficient of friction of 5 wt.% nano- ZrO_2 filled BMI nanocomposite is 0.62 that amounts to 82% of the neat BMI matrix. Detachment of the nanoparticles and small amount of the surrounding matrix plays the main role in the material removal due to wear. The detached nanoparticles might also act as solid lubricants. These account for the lower specific wear rates and friction coefficients of the nanocomposites.

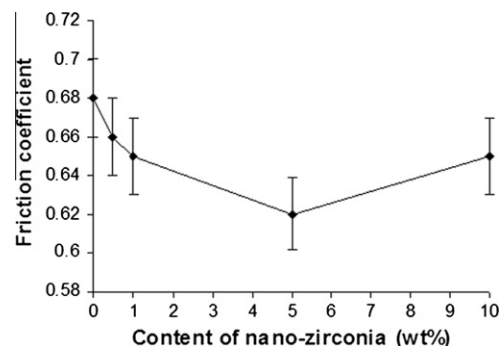


Fig. 6. Coefficient of friction of neat BMI and nano-zirconia filled BMI nanocomposites.

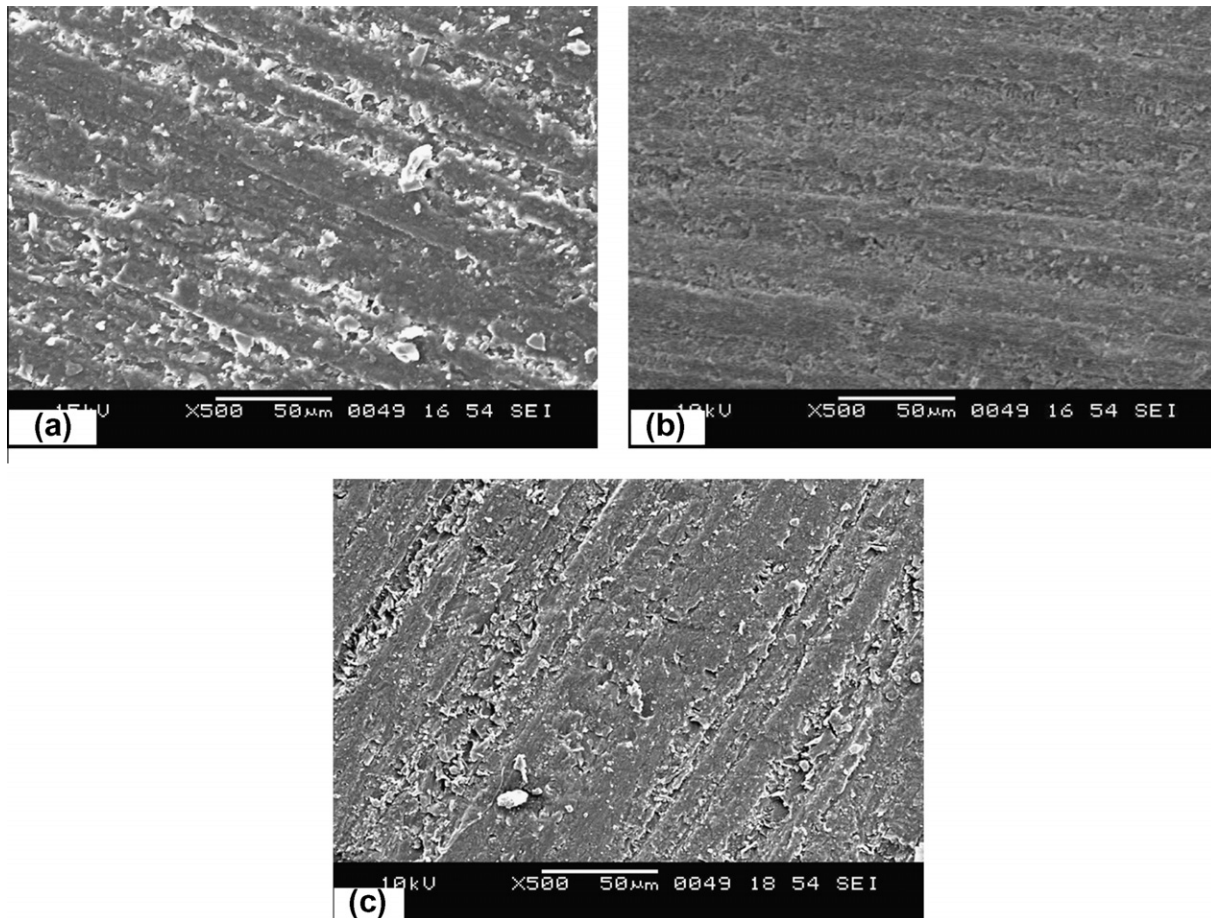


Fig. 7. Worn surface features of (a) neat BMI, (b) 5 wt.% nano-zirconia filled BMI, and (c) 10 wt.% nano-zirconia filled BMI nanocomposites.

3.3. Worn surface morphology

Observations of the worn surfaces by scanning electron microscope (SEM) provide knowledge about both, the role that nanoparticles play in the reduction of wear rate, and the wear mechanisms. Some wear pattern representatives for each sample are displayed in Fig. 7a–c.

Main features on the worn surface of the neat BMI resin are severe damage characterized by the disintegration of the top surface, wear debris and deep grooves in the sliding direction (Fig. 7a). Polymer fragments of the size of micrometers detach and leave small craters behind. These polymer fragments, which might be captured between the counterface and the pin sample, abrade the sample surface leading to even more substantial loss of material.

In the case of filled nanocomposites, the appearances are completely different and become rather smooth. Although the ploughing grooves are still visible on the nanocomposite sample surface, the groove depths are shallow on 5 wt.% filled BMI (Fig. 6) and at some region, the grooves are simply invisible (Fig. 7b). It seems the low filler loading is insufficient and cannot bring about significant improvements in wear resistance (Fig. 5). The wear performance of a composite material is indeed known to be volumetric dependent. It is noted that some cracks across the wear tracks are visible on the composites' worn surface (Fig. 7b). Very fine scratches are visible resulting from the nano-fillers pulled out of the matrix, and then further moved across the surface by scratching and rolling. Thus, it can be concluded that the abrasive wear of neat BMI is replaced by fatigue wear when the nanoparticles are

introduced. Due to their size, the particles should be able to move into gaps in the counterface and may then be exposed to rolling rather than sliding/scratching movement. Large matrix fragments are not found. During sliding, a rolling effect of nanoparticles could reduce the shear stress, the friction coefficient, and the contact temperature. The matrix damages in the interfacial region were reduced by this rolling effect. This process probably plays a key role to the reduced friction coefficient. Further, the ability of nano-ZrO₂ to improve the tribological behaviour is increased with decreasing the particle size of nanometer ZrO₂. The wear rate of larger particle size ZrO₂-filled PEEK was high because the composite transfer film had poor adhesion to the counterface [7]. On the other hand, in the present work, the wear-resistant BMI composite which was filled with small size ZrO₂ particles (<70 nm) transferred well to the counterface and its transfer film was thin, uniform and adhered strongly to the counterface. Thus the improvement in the tribological behaviour of nanometer ZrO₂-filled BMI composite is related to the improved characteristics of the transfer film. However, the wear resistance suffers if the filler loading exceeds 5 wt.% and the large amount of nanoparticles cannot provide any wear reducing effect. Abrasive wear is accompanied by delamination and fatigue cracking of the matrix (Fig. 7c).

4. Conclusions

This study focused on the development of nanocomposites with properties superior to the neat matrix. The following conclusions can be drawn:

- The addition of nano-ZrO₂ particles increases the hardness of BMI nanocomposites. The tribological performance of the composites showed a good correlation with the hardness up to 5 wt.% of filler loading.
- Based on microscopic observations of the worn surfaces, a positive rolling effect of the debris (matrix and nanoparticles) between the sample and the counterface, which led to remarkable reduction of friction coefficient. This effect becomes more remarkable when the nano-ZrO₂ content is 5 wt.%.
- The wear resistance of BMI nanocomposites considerably increases with increasing content of ZrO₂. The wear-resistant BMI composite which was filled with small size ZrO₂ particles (<70 nm) transferred well to the counterface and its transfer film was thin, uniform and adhered strongly to the counterface. Thus the improvement in the tribological behaviour of nanometer ZrO₂-filled BMI composite is related to the improved characteristics of the transfer film. An optimum wear resistant composition was found to be BMI with 5 wt.% nano-ZrO₂ particles.
- Nano-ZrO₂ filled BMI nanocomposites show signs of mild abrasive wear due to the hard ceramic particles. The main wear mechanism of composite changed from the severe abrasive wear (for neat BMI matrix) to mild abrasive wear.

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